

## **CONTINUOUS SURFACE PREPARATION OF METALS**

### **INVENTORS**

Kay Y. Blohowiak  
Shane E. Arthur  
William B. H. Grace  
Robert A. Anderson  
Matthew S. Tillman  
Darrin M. Hansen  
Steve R. Jones  
Rick G. Wire

### **RELATED PATENTS**

The related patents are U.S. Patent No. 5,814,137 issued September 29, 1998, U.S. Patent No. 5,849,110 issued December 15, 1998, U.S. Patent No. 5,869,141 issued February 9, 1999, U.S. Patent No. 5,939,197 issued August 17, 1999, U.S. Patent No. 5,958,578, and U.S. Patent No. 6,037,060 issued March 14, 2000, all of which are incorporated by reference herein.

### **RELATED APPLICATIONS**

This application is a continuation-in-part of U.S. Patent Application No. 10/143,391 filed May 9, 2002 and the benefit of that application's filing date is hereby claimed.

Additionally, this application incorporates by reference herein the application titled "Fiber-Metal Laminate Interphase Coating" invented by Matthew S. Tillman et al.; U.S. Patent Application No. 10/143,375 filed May 9, 2002.


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**BLACK LOWE & GRAHAM <sup>PLC</sup>**

  
701 Fifth Avenue, Suite 4800  
Seattle, Washington 98104  
206.381.3300 • F: 206.381.3301

## FIELD OF THE INVENTION

This invention relates generally to metal surface treatments and, more specifically, to a continuous surface preparation of metals.

## BACKGROUND OF THE INVENTION

Metal treatment prior to bonding is a key factor for both the initial adhesion of a bond and its long-term durability in aircraft, aerospace, and other applications. Current metal prebond surface preparations are either inconvenient or complex to use, contain hazardous materials (strong acids, hexavalent chromium, volatile organic compounds), and/or do not provide the performance necessary for successful long-term durable bonds. Past bond failures, primarily due to inadequate surface preparation, have been a limiting factor in the current use of bonded hardware, especially for primary structure.

In particular, the bonding of titanium using standard surface preparation techniques has not always been an easy or reproducible process. The very passive nature of titanium and the difficulty involved in chemical processing of titanium alloys have minimized the use of bonded titanium parts for primary or secondary structure. Several programs have used titanium bonding successfully; however, the surface preparation techniques employed are often arduous and involve hazardous chemicals and processes.

Therefore, there is an unmet need in the art for an economical and environmentally sound continuous surface preparation process for titanium and other metals such as aluminum, stainless steel, nickel, and copper prior to bonding with other materials.

## SUMMARY OF THE INVENTION

Embodiments of the present invention provide continuous surface preparation processes for titanium, aluminum, stainless steel, nickel, and copper. The processes of the present invention provide for surface treatment of metals in economical and environmentally-friendly manners.

According to an embodiment of the present invention, a continuous process removes oxides from titanium, aluminum, stainless steel, nickel, and copper by grit blasting the metal with a mixture of fine particles of aluminum oxide in air and water. The metal is rinsed with water to remove the grit from the metal.

In accordance with an aspect of the invention, a continuous process applies a sol-gel coating to a metal material by subjecting the material to a caustic solution of sodium hydroxide. The material is rinsed with water to remove the caustic solution of sodium hydroxide. A sol-gel coating is applied to the metal and the water portion of the sol-gel coating is evaporated.

In accordance with another aspect of the invention, a continuous process applies an adhesive coating on to a sol-gel coating on a metal material by applying a liquid adhesive coating to the sol-gel coating on the metal. The solvent portion of the adhesive coating is evaporated.

In accordance with yet another aspect of the invention, a continuous surface preparation process for metal material grit blasts the metal with a mixture of fine particles of aluminum oxide in air and water. The metal is rinsed with water to remove the grit. The metal material is subjected to a caustic solution of sodium hydroxide and the metal is rinsed with water to remove the caustic solution of sodium hydroxide. A sol-gel coating is applied to the metal and the water portion of the sol-gel coating is evaporated. A liquid adhesive coating is applied to the sol-gel coating on the metal and the solvent portion of the adhesive coating is evaporated.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

The preferred embodiments of the present invention are described in detail below with reference to the following drawings.

FIGURE 1 is a schematic illustration of a grit blast line;

FIGURE 2 is a schematic illustration of a sol-gel coating line; and

FIGURE 3 is a schematic illustration of an adhesive coating line.

#### **DETAILED DESCRIPTION OF THE INVENTION**

The present invention provides a continuous surface preparation process for metals such as titanium, aluminum, stainless steel, nickel, and copper.. The term "metals" as used herein includes alloys. The process of the present invention may be utilized in the overall process of forming titanium/graphite (TiGr) laminate structures. By way of overview and with reference to FIGURES 1, 2, and 3 one presently preferred embodiment of the present invention includes a process including a grit blast line 10 (FIG. 1), a sol-gel coating line 40 (FIG. 2), and an adhesive coating line 70 (FIG. 3). It is to be understood that it is an aspect of the present invention to combine the three separate lines 10, 40, and 70 into a single continuous line if desired. Specific details of the process are described with more detail below.

Metal material 14 in FIG. 1 is selected from the group of titanium, aluminum, stainless steel, nickel, and copper. For example, material 14 may be a foil of the titanium alloy Ti-15V-3Cr-3Al-3Sn, and is suitably from about 0.002 to about 0.016 inches thick. Grit blast line 10 performs grit blasting of both sides of material 14 inside wet hone chamber 22 which houses oscillating grit blaster nozzles which expel a mixture of fine particles of

aluminum oxide (alumina) in air and water. The grit has a mesh size of about 180-320 with a preferred mesh size of about 280. Material 14 is unwound from coil 12 and wound on coil 32. Material 14 travels through grit blast line 10 at a rate of about 5-10 feet per minute with a preferred rate of about 7.5 feet per minute. The wet hone process removes the oxide layer from both sides of material 14. Material 14 is tilted from a horizontal orientation to a vertical orientation by tilt rollers 16, 18, 20 before entering wet hone chamber 22.

A multiple stage water rinse is performed at ambient temperature in rinse chamber 24 to remove any grit on the material. An air curtain (not shown) at the exit side of chamber 24 blows high pressure air across both sides of material 14 to dry it. At the right end of line 10, material 14 is tilted back to a horizontal orientation from a vertical orientation by tilt rollers 26, 28, 30. Material 14 is then wound onto coil 32.

The coil is transported to sol-gel line 40 shown in FIG. 2. The coil is now designated as coil 34 for purposes of clarity. In FIG. 2, material 14 is unwound from coil 34 over rollers 36, 48, and 68 and wound on coil 70. Material 14 travels through sol-gel line 40 at a rate of about 3-7 feet per minute with a preferred rate of about 5 feet per minute. In caustic conditioner chamber 42, material 14 is subjected to a concentrated caustic solution of sodium hydroxide that is applied by upper spray heads 38 and lower spray heads 44. The concentration of the caustic solution is about 10-50% by weight sodium hydroxide with a preferred concentration of about 25% by weight sodium hydroxide. The temperature of the caustic solution is about 150-220°F with a preferred temperature of about 190°F. The purpose of the caustic conditioner is to form a controlled layer of metal oxy-hydroxide on both surfaces of material 14. The oxy-hydroxide layer is needed for sufficient chemical bonding to the subsequent sol-gel coating in chamber 54.

The caustic solution is washed off material 14 inside rinse chamber 46 using warm water applied by upper spray heads 50 and lower spray heads 52. Squeegees (not shown) inside rinse chamber 46 dry off the bulk of the rinse water. In sol-gel coating chamber 54, the water-based sol-gel solution is sprayed on material 14 by spray nozzles 56 and 60. The sol-gel materials are fully described in U.S. Patent Application No. 10/143,375 filed May 9, 2002, U.S. Patent No. 5,814,137 issued September 29, 1998, U.S. Patent No. 5,849,110 issued December 15, 1998, U.S. Patent No. 5,869,141 issued February 9, 1999, U.S. Patent No. 5,939,197 issued August 17, 1999, U.S. Patent No. 5,958,578, and U.S. Patent No. 6,037,060 issued March 14, 2000, all of which are incorporated by reference herein.

For example, the sol-gel may be an aqueous mixture of a zirconium alkoxide such as zirconium n-propoxide, 3-glycidoxy-propyltrimethoxysilane, glacial acetic acid, and a surfactant. The surfactant is suitably Antarox BL-240 made by Rhodia Inc. of Cranbury, NJ

08512-7500. However, other surfactants are considered within the scope of this invention, such as, without limitation, Tomadol 91-8 made by Tomah3 Products, Inc. of Milton, Wisconsin 53563-0388.

Metering rollers 58 and 62 skim off excess sol-gel solution on the surfaces of material 14. The wet sol-gel layer is about 1-5 mils thick before drying, with a preferred thickness of about 2.5 mils. The excess sol-gel solution falls into collecting pan 64. Material 14 travels into oven 66 where the water portion of the sol-gel is evaporated off. As dried, the sol-gel layer is about 10-500 nm thick, with a preferred thickness of about 100 nm. The temperature in oven 66 is about 150°F. Material 14 is wound on coil 70.

The coil is transported to adhesive line 100 shown in FIG. 3. The coil is now designated as coil 76 for purposes of clarity. In FIG. 3, material 14 is unwound from coil 76 over rollers 96, 88, and 90 and wound on coil 78. Material 14 travels into the bottom of two-part chamber 74. The lower part is the adhesive coating section and the upper part is an oven section 92. In the coating section, two methods of applying the adhesive are illustrated. One method is the adhesive dip-coating tank 98 shown in phantom at the bottom. The other method shown in FIG. 3 uses spray nozzles 82 and 84 to spray on the adhesive coating. Other methods of applying the adhesive may be employed such as graveure coating and reverse roller coating. The dry adhesive coating is also known as an interphase coating and has a thickness of 0.1 to 3.0 mils with a preferred thickness of 0.75 mils. The adhesive coating should have a uniform, consistent thickness.

The adhesive material is fully described in U.S. Patent Application No. 10/143,375 filed May 9, 2002 which is incorporated herein by reference. In a presently preferred embodiment, the epoxy-based adhesive coating includes a first epoxy material including approximately 25% solids by weight and a second curative material including approximately 32% solids by weight. The first epoxy material suitably includes about 3% to about 35% by weight liquid diglycidylether of bisphenol-A, about 35% to about 60% by weight solid diglycidylether of bisphenol-A, about 10% to about 30% by weight novolac-epoxy, and about 5% to about 18% by weight solid carboxy-terminated acrylonitrile-butadiene rubber, and the second curative material suitably includes about 0% to about 100% by weight 4,4'-diaminodiphenylsulfone, about 0% to about 100% by weight 3,3'-diaminodiphenylsulfone, and about 0% to about 0.2% by weight chromium octoate. An organic solvent, such as acetone, is used to dissolve the adhesive.

Material 14 travels upwardly through oven section 92 where the solvent is evaporated off. The temperature in oven 75 is about 150-170°F with a preferred temperature of about

160°F. Heated air is supplied to oven 75 through inlet duct 72 and the solvent vapor exits through exhaust duct 86.

As material 14 passes over roller 88, a backing film 96 from coil 94 is applied to material 14. Backing film 96 prevents the coated material from sticking to itself. The preferred backing film is thermoplastic film made of polyethylene terephthalate (PET) having a thickness of about 4 mils. Material 14 is wound on coil 78. Coil 78 has uncured epoxy material on the material surfaces, therefore it must be stored in a freezer until the further manufacturing steps (for example, laying down a composite layer and doing the lamination) are ready to be undertaken.

While the preferred embodiment of the invention has been illustrated and described, as noted above, many changes can be made without departing from the spirit and scope of the invention. Accordingly, the scope of the invention is not limited by the disclosure of the preferred embodiment. Instead, the invention should be determined entirely by reference to the claims that follow.